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## Indian Standard

# METHOD OF TEST FOR ACID-INSOLUBLE CONTENT IN IRON, COPPER, TIN AND BRONZE POWDERS

(First Revision)

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INDIAN STANDARDS INSTITUTION
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NEW DELHI 110002

## Indian Standard

## METHOD OF TEST FOR ACID-INSOLUBLE CONTENT IN IRON, COPPER, TIN AND BRONZE POWDERS

## (First Revision)

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## Indian Standard

## METHOD OF TEST FOR ACID-INSOLUBLE CONTENT IN IRON, COPPER, TIN AND BRONZE POWDERS

## (First Revision)

#### 0. FOREWORD

- **0.1** This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 29 November 1985, after the draft finalized by Powder Metallurgical Materials and Products Sectional Committee had been approved by the Structural and Metal Division Council.
- 0.2 This standard was first published in 1974. While reviewing the standard in the light of the experience gained during the years, the Sectional Committee decided to revise the standard. In this revision the test method for tin and bronze powders, have been included and the standard has been made in line with International Standard.
- **0.3** The insoluble matter referred to is generally considered to be acid-insolube sillica and sillicates, carbides, alumina, clays or other refractory oxides which are either present in the raw material from which the powders are manufactured or introduced during the manufacturing process.
- **0.4** In the formulation of this standard, assistance has been derived from ISO 4496-1978 'Metallic powders Determination of acid insoluble content of iron copper tin and bronze powder,' issued by the International Organization for Standardization (ISO).
- 0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960\*.

#### 1. SCOPE

1.1 This standard prescribes the method for the determination of the mineral acid insoluble matter content of iron, copper, tin and bronze powders.

<sup>\*</sup>Rules for rounding off numerical values ( revised ).

1.2 The method is applicable to lubricant-free powder.

#### 2. PRINCIPLE OF TEST

2.1 The test consists of dissolving the powder in appropriate acid. The insoluble matter is filtered out, washed and ignited in a furnace at 980 + 20°C.

#### 3. SAMPLING

3.1 The sampling shall be done in accordance with IS: 6492-1972\*.

#### 4. QUALITY OF REAGENTS

4.1 During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

## 5. DETERMINATION OF ACID-INSOLUBLE MATTER OF IRON POWDER

#### 5.1 Reagents

**5.1.1** Hydrochloric Acid — 1:1 and 1:25 (v/v).

#### 5.2 Procedure

5.2.1 Weigh a 5 g sample of the powder to the nearest 1 mg and transfer to a glass beaker. Carefully add 100 ml of hydrochloric acid (1:1) and let it stand at room temperature until the reaction is complete (no further evolution of hydrogen).

Note — If it is desired to exclude carbides from the reported insoluble matter, add 20 ml of concentrated nitric acid to hydrochloric acid (1:1).

- **5.2.2** Heat the solution on a hot plate to boiling. Add 150 ml of water and reheat to boiling and maintain for about 1 minute. Allow the solution to cool and settle for 5 minutes.
- 5.2.3 Filter the solution through a medium filter paper (for example, Whatman 40) and wash the residue alternately with hot hydrochloric acid (1:25) and hot water. Repeat the washing until the residue is free of iron salts.
- 5.2.4 Transfer the filter paper and residue to a fused silica or porcelain crucible which had been preheated to constant mass at  $980 \pm 20^{\circ}$ C and weighted to the nearest 0·1 mg. Ignite the residue in a furnace at a temperature  $980 \pm 20^{\circ}$ C for one hour. Cool in a desiccator and re-weigh. Ignite again and ensure that the difference between two consecutive weighings of the crucible with the residue after cooling is not greater than 0·1 mg.

<sup>\*</sup>Methods for sampling of powders for powder metallurgical purposes.

**5.3 Calculation** — The acid insoluble content (percent by mass) = 
$$\frac{m_3 - m_2}{m_1} \times 100$$

where

 $m_1 = \text{mass in g of the sample,}$ 

 $m_2 = \text{mass in g of empty crucible, and}$ 

 $m_3 =$ mass in g of the crucible with residue.

## 6. DETERMINATION OF ACID-INSOLUBLE CONTENT OF TIN, COPPER AND BRONZE POWDERS

#### 6.1 Reagents

- **6.1.1** Dilute Nitric Acid 1:1 (v/v).
- **6.1.2** Concentrated Hydrochloric Acid rd = 1.16 (conforming to IS: 265-1976\*).
  - **6.1.3** Ammonium Acetate Solution -20 percent (w/v).

#### 6.2 Procedure

- 6.2.1 Weigh a 5 g sample of the powder to the nearest 1 mg and transfer it to a glass beaker carefully. Add 50 ml of concentrated hydrochloric acid. Cover it with a watch glass and digest at low temperature for a minimum of 30 minutes by placing the beaker on a hot sand bath.
- 6.2.2 Remove the beaker, and cool it at room temperature. Add carefully 50 ml of dilute nitric acid. Wait for the initial reaction, which starts in about 10 minutes. After the initial reaction is complete, add 50 ml of more dilute nitric acid. Boil the solution until the volume is reduced to one half. Gradually add 50 ml of hot water and boil for about 1 minute. Allow the solution to cool and settle for 5 minutes.
- 6.2.3 Filter the solution through a medium filter paper (for example, Whatman 40) and wash the residue first with hot concentrated hydrochloric acid and finally with hot water. If the presence of lead salt is suspected, wash at least twice with a hot ammonium acetate and then with water.
- 6.2.4 Repeat the washing with water until copper or tin salts are not detected in the washing (copper can be detected by 4 percent sodium diethyl dithio carbamate solution and tin may, be detected by hydrogen sulphide or sodium sulphide).

<sup>\*</sup>Specification for hydrochloric acid ( second revision ).

**6.2.5** Transfer the filter paper with residue to a fused silica or porcelain crucible which had been preheated to constant mass at  $980 \pm 20^{\circ}\mathrm{C}$  and weighed to the nearest 0·1 mg. Place the crucible on a hot plate to dry and char the paper. Ignite in the furnace at a temperature  $980 \pm 20^{\circ}\mathrm{C}$  for one hour. Cool in a desiccator and reweigh. Ignite again and ensure that the difference between two consecutive weighings of the crucible with residue after cooling is not greater than 0·1 mg.

#### 6.3 Calculation

The acid soluble content, (percent by mass) = 
$$\frac{m_3 - m_2}{m_1} \times 100$$

where

 $m_1 = \text{mass in g of the sample,}$   $m_2 = \text{mass in g of empty crucible, and}$  $m_3 = \text{mass in g of the crucible with residue.}$ 

6.4 Calculate the result of each determination to the nearest 0.01 percent.

#### 7. TEST REPORT

- 7.1 The test report shall include the following information:
  - a) reference to this standard;
  - b) all details necessary for identification of the test sample;
  - c) the result obtained; and
  - d) details of any occurrence which may have affected the test result.



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